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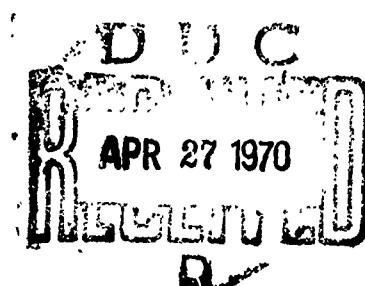
AFRPL-TR-69-219

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# (U) THE SYNTHESIS OF NEW IONIC INTERHALOGEN OXIDIZERS

F. Q. ROBERTO

OCTOBER 1969



## TECHNICAL REPORT AFRPL-TR-69-219

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AFRPL-TR-69-219

THE SYNTHESIS OF NEW IONIC  
INTERHALOGEN OXIDIZERS (U)

Francisco Q. Roberto

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FOREWORD

(U) This is a phase report of work conducted at the Air Force Rocket Propulsion Laboratory to determine the feasibility of producing interhalogen high-energy liquid oxidizers. This work was accomplished under Project 314801ACL during the period of July 1968 through July 1969 by Dr. Francisco Q. Roberto, Task Scientist. The author wishes to acknowledge the valuable technical discussions and support furnished by Dr. Claude Merrill, Project Scientist; Dr. Charles Bock for X-ray diffraction work and to Mr. John H. Leahy for the analysis of this hazardous and very reactive material. The author also wishes to acknowledge Dr. George Begun, Oak Ridge National Laboratory for running the Raman spectral study.

(U) This report has been reviewed and approved.

NORMAN J. VANDER HYDE  
Chief, Solid Propellant Branch  
Propellant Division  
Air Force Rocket Propulsion Laboratory

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CONFIDENTIAL ABSTRACT

(C) The synthesis of hexafluorochloronium (VII) hexafluoroplatinate (V),  $\text{ClF}_6^+\text{PtF}_6^-$ , from the reaction of chlorine pentafluoride and platinum hexafluoride is described as an oxidation-reduction reaction. This has been confirmed by elemental analyses, infrared and Raman Spectroscopy, X-ray powder diffraction and displacement reaction with trifluorochlorine oxide. This is the first example of a perfluorinated heptavalent chlorine cation. The compound,  $\text{ClF}_6^+\text{PtF}_6^-$ , has been indexed as having a cubic structure based on preliminary X-ray powder pattern.

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## **SECTION I**

### **INTRODUCTION**

(U) Reactions of platinum hexafluoride with other oxidizing agents have been studied by several investigators.<sup>(1-6)</sup> With chlorine trifluoride, difluorochloronium (III) hexafluoroplatinate (V) ( $\text{ClF}_2\text{PtF}_6$ ), and chlorine pentafluoride<sup>(7)</sup> are produced.<sup>(6)</sup> Gortsema and Toeniskoetter<sup>(6)</sup> also attempted to react platinum hexafluoride with chlorine pentafluoride; the only product observed, in addition to the reactant, was a yellow solid believed to be  $\text{ClF}_2\text{PtF}_6$  (it was assumed that it resulted from the decomposition of chlorine pentafluoride into chlorine trifluoride and subsequent reaction of chlorine trifluoride with platinum hexafluoride). Recent studies of the same reaction have shown that tetrafluorochloronium (V) hexafluoroplatinate (V) was produced.<sup>(8)</sup> Other salts of tetrafluorochloronium ion have been reported recently.<sup>(9)</sup>

(C) Studies in this laboratory of platinum hexafluoride with chlorine pentafluoride have shown that in an excess of chlorine pentafluoride using a sapphire reactor, a solid product, believed to be hexafluorochloronium (VII) hexafluoroplatinate (V) ( $\text{ClF}_6^+\text{PtF}_6$ ), was obtained. The cation  $\text{ClF}_6^+$  has been reported previously.<sup>(10, 11)</sup> However, most of the evidence was based on infrared data and more recent studies have disputed its synthesis.<sup>(12)</sup> The solid produced in this laboratory is markedly different from those reported<sup>(10-12)</sup> and new data are presented for the synthesis of  $\text{ClF}_6^+$  in this laboratory.

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SECTION II

EXPERIMENTAL PROCEDURES

A. EQUIPMENT

(U) The vacuum system used in this work consisted of a nickel manifold constructed from 1/4-inch nickel pipe to which a number of subsystems were attached. These were: (1) a multipurpose line for handling, measuring, and transferring reactants; (2) a line for purifying and storing fluorine; and (3) a similar line for purifying halogen fluorides. All valves (Hoke TM 413 monel diaphragm valves with metal seats) were silver-soldered to the manifold outlets. Pressure measurements were made with the Wallace and Tiernan gage, Model FA 145, accurate to 0.2 torr. Volumes were calibrated using pure helium and nickel bulbs of known volume.

(U) The vacuum system and sapphire reactors were passivated with fluorine at 300° using a heat gun. Passivation was considered complete when pressure at ambient temperature was identical before and after heating to 300°. Before each run, the whole system was passivated with chlorine pentafluoride followed by a final passivation with platinum hexafluoride.

B. MATERIALS

(U) Platinum hexafluoride was obtained from Ozark-Mahoning Company, Tulsa, Oklahoma. The material was further purified by low-temperature distillation according to the procedure described.<sup>(1)</sup>

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The infrared spectrum<sup>(13)</sup> and vapor pressure of the pure material agreed well with the published values.

(C) Chlorine pentafluoride and trifluorochlorine oxide, ClF<sub>3</sub>O,<sup>(12)</sup> were obtained from Rocketdyne, a Division of North American Aviation, Canoga Park, California. The material was used without further purification.

#### C. ANALYSIS

(U) The platinum compounds were transferred into a 50-ml quartz reactor that had been previously weighed in the dry box and purged with dry nitrogen. The reactor was then connected to the vacuum system to remove the nitrogen and reweighed. The sample was frozen at -196° and 5-10 ml of double-distilled water was transferred to the sample. Immediate reaction was accompanied by a flash when vapor initially came into contact with the sample. The sample was warmed to ambient temperature by removing the liquid nitrogen trap. After half an hour at ambient temperature, the solution was refrozen to -196° and 5-10 ml of hydrazine was transferred to it. The solution was again warmed to ambient temperature. The platinum solids formed by hydrolysis and reduction were filtered off; they were redissolved with aqua regia to convert the platinum-containing materials to chloroplatinic acid. The solution was diluted with water, reduced with sodium formate,<sup>(14)</sup> and the finely divided platinum metal was filtered, dried, and weighed. After additional dilution, aliquots of the stock solution were analyzed for chlorine and fluorine. Chloride ions were determined by anodic chronopotentiometry using a silver electrode.

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Fluoride ion was determined potentiometrically with an Orion fluoride ion electrode.<sup>(15)</sup> The above methods were checked with standard samples.

(U) X-ray diffraction studies were carried out using the powder techniques with samples sealed in 0.3-mm thin-wall quartz capillary tubes. The tubes were previously flamed in a vacuum line to remove moisture. Samples were loaded in a dry box, flushed with dry nitrogen, sealed with Kel-F grease, and placed in a 5.73-cm Debye-Scherrer camera. Measurements were made using copper  $K\alpha_1$  radiation with a nickel filter. Exposure times varied from 1/2 hour to 4 hours. Intensities were estimated visually.

(U) Infrared spectra were obtained with Beckman IR-5A and Perkin-Elmer 337 spectrophotometers. Gaseous spectra were obtained using a 10-cm nickel cell equipped with silver chloride windows. Spectra were obtained on the solid pressed between chloride windows of the nickel infrared cell. All operations were carried out in a dry box.

#### D. REACTION OF PLATINUM HEXAFLUORIDE WITH CHLORINE PENTAFLUORIDE

(C) In a typical reaction, 1.26 mmole (0.390 grams) of platinum hexafluoride, and 3.78 mmole (0.491 grams) of chlorine pentafluoride were condensed at -196° into an evacuated, prepassivated 7-cc sapphire reactor. The reaction mixture was slowly warmed to ambient temperature. The mixture was left exposed to light at 22-23° for a period of 8 days. The reaction was monitored by the disappearance of  $PtF_6$  (red gas). As time progressed, the red-brown solid phase became lighter. After 8 days,

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the solid turned to the bright yellow color of the  $\text{ClF}_x\text{PtF}_6$  salt. The reactor was cooled to -196° and found to contain no noncondensable gas. The gaseous mixture was found to contain only  $\text{ClF}_5$ .<sup>(16)</sup> The excess  $\text{ClF}_5$  was removed under vacuum giving a bright orange-yellow solid with no vapor pressure at room temperature. The weight of the solid product was 0.489 grams. The material was analyzed according to the procedure described.

(C) Anal. Calcd. for  $\text{ClF}_6\text{PtF}_6$ : Pt, 42.56; F, 49.7; Cl, 7.73.  
Found: Pt, 42.7; F, 47.2; Cl, 5.2.

(C) The infrared spectrum of the yellow solid was prepared in the dry box by pressing it between silver chloride windows. This is shown in Figure 1. The X-ray powder pattern of the solid is given in Table I.

#### E. REACTION WITH $\text{ClF}_3\text{O}$

(C) In order to further characterize the  $\text{ClF}_x\text{PtF}_6$ , excess  $\text{ClF}_3\text{O}$  was condensed on top of the solid of  $\text{ClF}_x\text{PtF}_6$  at -196°C. The reactor was gradually warmed to -85°C using trike-liquid nitrogen slush. There was very little evidence of reaction at this temperature for 30 minutes. At -75°C, there was some reaction and the only volatile product found was  $\text{ClF}_5$  and a trace of  $\text{ClFO}_2$ . This reaction continued up to -35°C producing more  $\text{ClF}_5$ . The total noncondensable (-196°C) was 5 mm after about 2 1/2 hours. The reaction was left overnight starting at -35°C and gradually warming to room temperature. The 40 millimeters of noncondensable at -196°C was identified (by mass spectral analysis and reaction

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with mercury) as fluorine. The  $\text{ClF}_5$  formed was transferred and weighed by pumping on the reactor at  $-85^\circ\text{C}$ . After all the volatile product was removed, the solid was weighed and subjected to infrared analyses. The infrared spectra was identical to the spectra of  $\text{ClOF}_2\text{PtF}_6^{(17)}$  previously obtained between the reactions of  $\text{ClF}_3\text{O}$  and  $\text{PtF}_6$ .

(\*) Based on the weight of fluorine, chlorine pentafluoride and  $\text{ClF}_2\text{OPtF}_6$  produced, the  $\text{ClF}_x\text{PtF}_6$  was calculated to contain a 50:50 mixture of  $\text{ClF}_4\text{PtF}_6$  and  $\text{ClF}_6\text{PtF}_6$  within experimental error. These results definitely established the presence of  $\text{ClF}_6^+$  in the reaction product. The reaction to produce the heptavalent  $\text{ClF}_6^+$  can be written as:



In excess  $\text{ClF}_5$ ,  $\text{PtF}_5$  reacts with it to give  $\text{ClF}_4\text{PtF}_6$ .

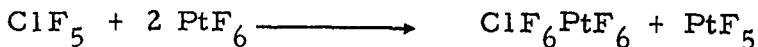
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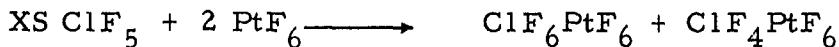
SECTION III

RESULTS AND DISCUSSION

(C) The solid product obtained in the reaction between platinum hexafluoride and chlorine pentafluoride using a sapphire reactor activated with light is believed to be  $\text{ClF}_6\text{PtF}_6$ , in accordance with the equation

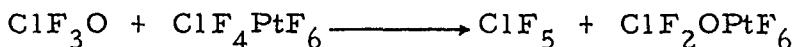


In excess  $\text{ClF}_5$ , the reaction proceeds further to give  $\text{ClF}_4\text{PtF}_6$ . The overall reaction in excess  $\text{ClF}_5$  can be expressed as:



The reaction can be described as an oxidation-reduction where chlorine is oxidized to the heptavalent state forming an ionic solid  $\text{ClF}_6^+\text{PtF}_6^-$  and  $\text{PtF}_5$ . However, in an excess  $\text{ClF}_5$ ,  $\text{PtF}_5$  is acting as a Lewis acid abstracting a fluoride ion from  $\text{ClF}_5$  to give  $\text{ClF}_4\text{PtF}_6$ .

(C) Further evidence for the synthesis of  $\text{ClF}_6\text{PtF}_6$  was obtained from the reaction with trifluorochlorine oxide. The mass balance obtained from the reaction of  $\text{ClF}_4\text{PtF}_6$ ,  $\text{ClF}_6\text{PtF}_6$  with  $\text{ClF}_3\text{O}$  indicates that  $\text{ClF}_4\text{PtF}_6$  reacted with  $\text{ClF}_3\text{O}$  at  $-75^\circ$  to  $-35^\circ$  according to the equation:



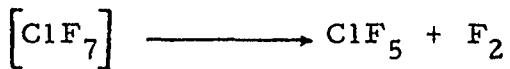
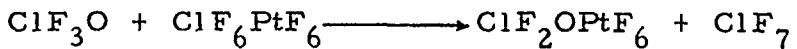
Above  $-35^\circ$ , melting point of  $\text{ClF}_3\text{O}$ ,  $\text{ClF}_6\text{PtF}_6$  then reacted with  $\text{ClF}_3\text{O}$  in one of two ways:

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or



Since the only volatile products observed at ambient temperature were  $\text{ClF}_5$  and fluorine, it is implied that  $\text{ClF}_7$  formed and is unstable at this temperature or it is catalytically decomposed by  $\text{ClF}_2\text{OPtF}_6$ . From this study the composition of the mixture was deduced to be a 50:50 mixture of  $\text{ClF}_6\text{PtF}_6$  and  $\text{ClF}_4\text{PtF}_6$  based on the weight of fluorine,  $\text{ClF}_5$  and  $\text{ClF}_2\text{OPtF}_6$  produced from the above equation.

(C) The infrared spectrum of solid mixture  $\text{ClF}_6\text{PtF}_6 + \text{ClF}_4\text{PtF}_6$  pressed between silver chloride plates consists of bands attributed to  $\text{ClF}_4\text{PtF}_6$  and bands at 889, 875 and  $540 \text{ cm}^{-1}$  assigned to  $\text{ClF}_6^+$  and the peak at  $649 \text{ cm}^{-1}$  attributed to the  $\text{PtF}_6^-$  absorption band. The 890, 875 and  $540 \text{ cm}^{-1}$  observed in the infrared were absent in the Raman spectrum as expected of an octahedral molecule. Since  $\text{ClF}_6^+$  is isoelectronic with the octahedral,  $\text{SF}_6$ , some similarity in their spectra is expected, except for slight frequency shifts due to their mass difference. The higher mass of the central atom in  $\text{ClF}_6^+$  compared to  $\text{SF}_6$  should cause a shift toward lower frequencies, as is indeed observed for the infrared active bands.

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Going from  $SF_6$  ( $940\text{ cm}^{-1}, \gamma_3$ ) to  $C1F_6^+$  ( $890\text{ cm}^{-1}, \gamma_3$ ), the shift is  $50\text{ cm}^{-1}$ ;  $\gamma_4$  for  $SF_6$  at  $615\text{ cm}^{-1}$  is shifted by  $65\text{ cm}^{-1}$  in  $C1F_6^+$  ( $540\text{ cm}^{-1}$ ).

These assignments are tentative and require further refinements.

(C) Of the six normal modes of vibration expected of an octahedral ion of the type  $XY_6$ , ( $A_{1g} + E_g + 2 F_{1u} + F_{2g} + F_{2u}$ ), only two of these modes ( $2 F_{1u}$ ) will be infrared active and three will be Raman active ( $A_{1g}$ ,  $E_g$ , and  $F_{2f}$ ).

(C) The remaining  $F_{2u}$  mode is inactive in both infrared and Raman spectrum. However, since the octahedral  $PtF_6^-$  mode of vibrations have not been assigned, it is rather difficult to assign vibrations due to  $C1F_6^+$  with any certainty. Since the only absorption band that we can assign with any degree of certainty to  $PtF_6^-$  is the absorption band at  $645\text{ cm}^{-1}$ , the other absorption bands, in addition to 890, 875 and 540, at 521 and 313  $\text{cm}^{-1}$ , may be attributed to  $C1F_6^+$ .

(C) The X-ray powder pattern for  $C1F_6PtF_6$  (after subtracting the lines due to  $C1F_4PtF_6$ ) is relatively simple, indicating high symmetry. The high symmetry of the unit cell of  $C1F_6PtF_6$  (probably cubic) seems reasonable since both  $C1F_6^+$  and  $PtF_6^-$  have octahedral symmetries. The only other known compound of the  $XF_6^+ YF_6^-$  structure is  $IF_6^+ SbF_6^-$ .<sup>(18)</sup> This salt has a high symmetry (face-centered cubic) and  $C1F_6PtF_6$  is expected to exhibit similar structural symmetry.

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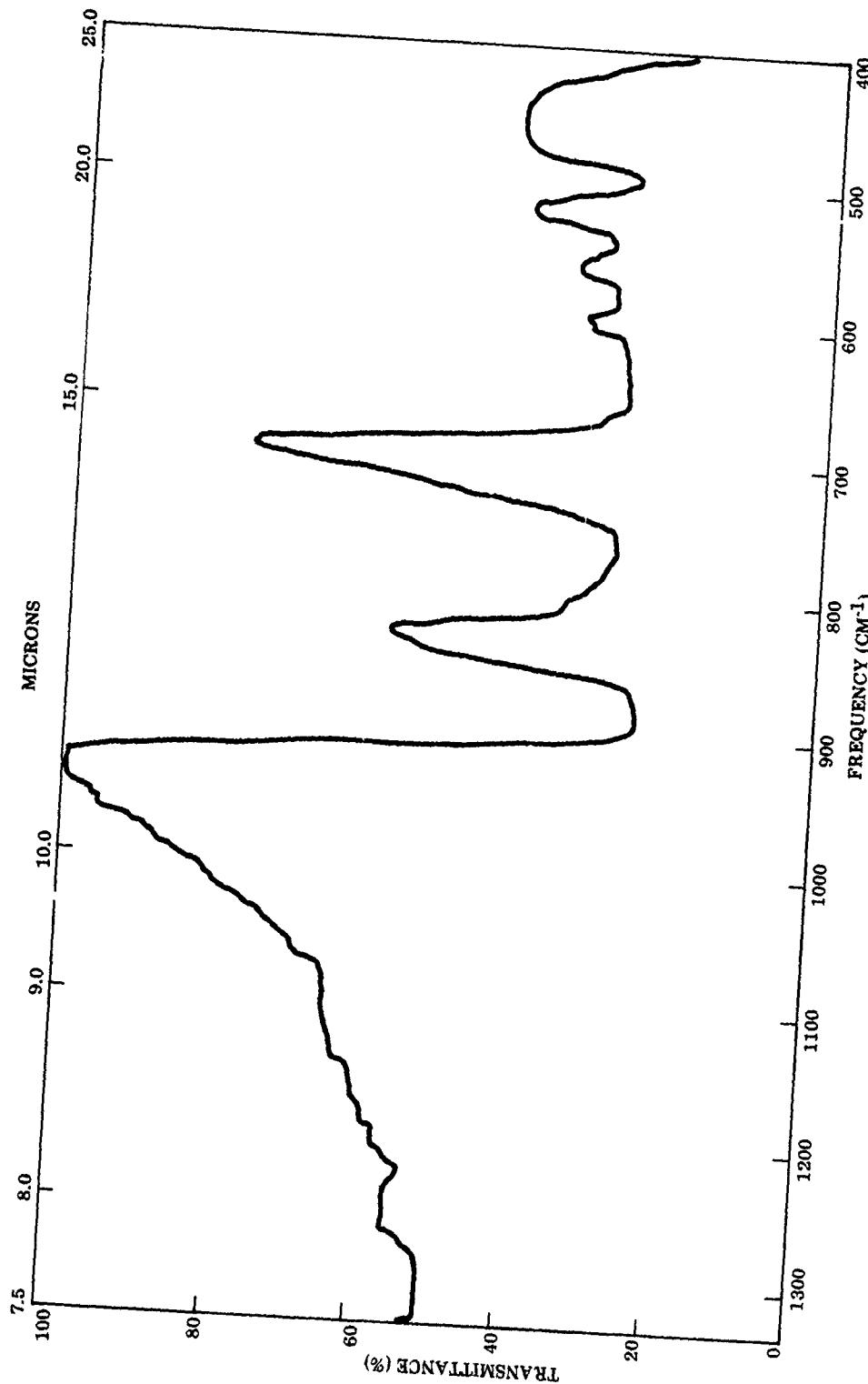


Figure 1. Infrared Spectrum of the Mixture of  $\text{ClF}_6^+\text{PtF}_6^-$  and  $\text{ClF}_4^+\text{PtF}_6^-$

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TABLE I. (C) X-RAY POWDER DATA FOR  $\text{ClF}_6^+ \text{PtF}_6^-$  \*

$d_{(\text{obsd})}$	$d_{(\text{calcd})}$	$hkl$	Intensity
5.717	5.74	110	Strong
4.036	4.06	200	Very Strong
3.328	3.32	211	Medium
2.576	2.569	310	Light
2.334	2.345	222	Medium
1.999	2.030	400	Light
1.923	1.915	411	Very Light
1.774	1.772	421	Very Light
1.727	1.732	332	Very Light
1.530	1.535	520, 432	Very Light

\* Tentatively identified as having a cubic symmetry.

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Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Air Force Rocket Propulsion Laboratory Edwards, California 93523	2a. REPORT SECURITY CLASSIFICATION Confidential
	2b. GROUP 4

3 REPORT TITLE

The Synthesis of New Ionic Interhalogen Oxidizers (U)

4 DESCRIPTIVE NOTES (Type of report and inclusive dates)

Phase Report July 1968 - July 1969

5. AUTHOR(S) (First name, middle initial, last name)

Roberto, Francisco Q.

6. REPORT DATE October 1969	7a. TOTAL NO OF PAGES	7b. NO OF REFS 18
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8a. CONTRACT OR GRANT NO

9a. ORIGINATOR'S REPORT NUMBER(S)

b. PROJECT NO 3148

AFRPL-TR-69-219

c.

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

d.

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11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

Air Force Rocket Propulsion Laboratory  
Air Force Systems Command, USAF  
Edwards, California

13 ABSTRACT

(C) The synthesis of hexafluorochloronium (VII) hexafluoroplatinate (V),  $\text{ClF}_6^+ \text{PtF}_6^-$ , from the reaction of chlorine pentafluoride and platinum hexafluoride is described as an oxidation-reduction reaction. This has been confirmed by elemental analyses, infrared and Raman spectroscopy, X-ray powder diffraction and displacement reaction with trifluorochlorine oxide. This is the first example of a perfluorinated heptavalent chlorine cation. The compound,  $\text{ClF}_6^+ \text{PtF}_6^-$ , based on preliminary X-ray powder pattern, has been indexed as having a cubic structure.

DD FORM 1 NOV 65 1473

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Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Heptavalent chlorine Hexafluorochloronium Interhalogen						

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